



EDGEWOOD

CHEMICAL BIOLOGICAL CENTER

U.S. ARMY RESEARCH, DEVELOPMENT AND ENGINEERING COMMAND

ECBC-TR-622

RESIDUAL LIFE INDICATOR FOR PHYSICAL ADSORPTION CAPACITY OF NBC FILTERS

PART I. ACETONE VAPOR PULSES AND THE EFFECT OF MOISTURE CONTENT ON RETENTION CHARACTERISTICS

Gregory W. Peterson

RESEARCH AND TECHNOLOGY DIRECTORATE

David Friday
Marc Shrewsbury

HUNTER
Manufacturing Company
A business unit of Hunter Defense Technologies

HUNTER APPLIED RESEARCH CENTER
Edgewood, MD 21040

June 2008

Approved for public release;
distribution is unlimited.



ABERDEEN PROVING GROUND, MD 21010-5424

20080721 202

Disclaimer

The findings in this report are not to be construed as an official Department of the Army position unless so designated by other authorizing documents.

Blank

PREFACE

The work described in this report was authorized under Job Order No. 5E22A. This work was started in January 2006 and completed in June 2006.

The use of either trade or manufacturers' names in this report does not constitute an official endorsement of any commercial products. This report may not be cited for purposes of advertisement.

This report has been approved for public release. Registered users should request additional copies from the Defense Technical Information Center; unregistered users should direct such requests to the National Technical Information Service.

Acknowledgment

The authors thank Jennifer Keller for her technical contributions in performing the experimental work.

Blank

CONTENTS

1.	INTRODUCTION	1
2.	BACKGROUND	1
2.1	Current RLI Methodology	1
2.2	ESLI versus RLI	2
2.3	RLI Theory.....	2
2.4	Historical Attempts at RLI Development	4
2.5	Tracer Development Approach.....	5
3.	EXPERIMENTAL PROCEDURES	6
4.	RESULTS AND DISCUSSION	7
4.1	Preliminary Testing.....	7
4.2	Life-Thickness Breakthrough Testing	9
4.3	Life-Thickness Pulse Testing.....	11
4.4	RH and Moisture Content Breakthrough Testing	12
4.5	RH and Moisture Content Pulse Testing	15
5.	CONCLUSIONS.....	19
	LITERATURE CITED	21

FIGURES

1.	Physical Adsorption Capacity Effect on Retention Time of Chemical Tracer	3
2.	Tracer Pulse and Agent Breakthrough of Contaminated Filters	4
3.	Methodology for Proof-of-Concept Tracer Testing.....	5
4.	Saturator Cell Used for Feed Delivery.....	7
5.	Acetone Breakthrough of ASZM-T at Various RH Conditions	8
6.	Acetone Breakthrough of ASZM-T at Various Bed Depths.....	9
7.	Acetone Breakthrough of ASZM-T at Various Bed Depths.....	10
8.	Acetone Breakthrough Time as a Function of ASZM-T Bed Depth	10
9.	Acetone Pulses to ASZM-T at Various Bed Depths.....	11
10.	Acetone Pulses to ASZM-T at Various Bed Depths.....	12
11.	Acetone Effluent Peak Height as a Function of ASZM-T Bed Depth.....	13
12.	Acetone Pulse Retention Time as a Function of ASZM-T Bed Depth.....	13
13.	Acetone Breakthrough Time as a Function of RH.....	14
14.	Acetone Breakthrough Time through ASZM-T as a Function of RH	14
15.	Acetone Pulse Retention as a Function of RH.....	15
16.	Acetone Pulse Retention as a Function of 30% RH	16
17.	Acetone Pulse Retention as a Function of 50% RH	16
18.	Acetone Pulse Retention as a Function of 65% RH	17
19.	Acetone Pulse Retention as a Function of 80% RH	18
20.	Elution Peak Height of Acetone Pulses as a Function of RH	18
21.	Acetone Pulse Retention Time as a Function of RH	19

TABLES

1.	Tracer Properties.....	6
2.	Pulse Test Conditions	6

RESIDUAL LIFE INDICATOR FOR PHYSICAL ADSORPTION CAPACITY OF NBC FILTERS

PART I. ACETONE VAPOR PULSES AND THE EFFECT OF MOISTURE CONTENT ON RETENTION CHARACTERISTICS

1. INTRODUCTION

Activated carbon, specifically ASZM-TEDA, is prevalent in most chemical, biological, radiological, and nuclear (CBRN) filters. This material possesses high adsorption capacities for physically adsorbing chemicals and is therefore an ideal sorbent for chemical warfare agent (CWA) filtration. However, because of its high adsorption capacity, environmental contaminants such as heavy organic molecules are also adsorbed on the filter. Depending on their strength of adsorption, these contaminants may affect CWA filtration due to either competition or blockage of micropores within the activated carbon structure. Currently, no method exists to indicate when CBRN filters no longer have the capacity to protect the warfighter in the event of a toxic chemical release. The following factors are responsible for CBRN filter failure:

- a. Loss of filter integrity, such as from a mechanical leak or adsorbent settling.
- b. Loss of physical adsorption capacity due to environmental contamination.
- c. Loss of reactive capacity due to impregnant migration and/or deactivation.

Financial and logistical concerns necessitate the development of a filter integrity tester (FIT) and residual life indicator (RLI). Current systems may be subject to certain change-out criteria when, in fact, the filter is still mechanically fit with remaining capacity. Conversely, a filter may have gone through a physical change due to temperature, the presence of water, or any number of possible circumstances that demand immediate change-out. The presence of an RLI integrated into a filter system allows the user to quickly assess the need for change-out without HVAC downtime, filter damage, or excessive financial loss.

This report explores the possibility of using acetone as an RLI for the residual adsorptive capacity of an in-service filter. The objective of this report is to determine the effects of RH and ASZM-TEDA bed depth on the retention characteristics of acetone pulses.

2. BACKGROUND

2.1 Current RLI Methodology

The current method for determining whether or not a collective protection (ColPro) filter has remaining capacity constitutes the removal of the filter and subsequent dimethyl methylphosphonate (DMMP) breakthrough testing on the carbon contained within the filter. This method, although effective in determining the residual life of that filter and possibly of the air purification system from which that filter was pulled, is destructive; therefore, the filter

must be replaced. As such, it would be beneficial to develop a method for non-destructive determination of the residual adsorption capacity of an in-place filter.

2.2 ESLI versus RLI

Two main schools of thought are prevalent in the search to find a solution to determining whether or not a filter has remaining capacity without destroying it: end-of-service-life indicators (ESLI) and RLI. End-of-service-life indicators are grouped as either active or passive systems. Active systems are typically monitored electronically; whereas, passive systems typically require user monitoring (Favas, 2005). Several ESLI technologies are prevalent today. One such method includes colorimetric sensors, which include paper strips and thin microfilms that change color when exposed to certain chemical compounds. Gardner et al. completed a study on colorimetric sensors in 2003. The main shortcoming of this technique is that in a ColPro environment, the sensor is continuously exposed to contaminants, possibly resulting in either early failure or false positives. Another ESLI technique more applicable to ColPro are probe-in-bed sensors (Mix and McDonald, 1990), which, as the name implies, are sensors that are embedded into filter beds at specific points to detect a chemical adsorption wave moving through the bed. However, the shortcoming of this type of ESLI technology to date is that the quantity and relative adsorption strength of the contaminant loaded on the carbon can not be determined. In other words, an ESLI may sense a contaminant moving through the gas filter; however, this contaminant may be a light chemical that would not affect agent filtration performance. As such, false alarms can lead to premature filter change-out and increased cost. Other ESLI methods employ similar concepts.

2.3 RLI Theory

On the other hand, RLIs are capable of distinguishing between the amount of contaminant on the filter as well as the relative strength of adsorption of that contaminant. Determining the remaining physical adsorption capacity of a filter can be accomplished by pulsing that filter with a known amount of chemical tracer and analyzing retention characteristics (e.g., peak height, shape, and retention time). The proposed technology uses a vapor pulse technique in which a chemical vapor (tracer) is delivered to a carbon bed under normal operating conditions. A detector is used to monitor the dosage of chemical delivered to the filter and that which may be detected downstream of the filter. The tracer's role is either to identify mechanical defects within the system or to provide information reflecting a change in the capacity (or state of activity) of the adsorbent to remove CWAs and toxic industrial chemicals (TICs). Application of a small vapor pulse of a highly volatile chemical to the inlet of the filter can provide much information as to the condition of the filter with respect to the three causes of filter failure. Figure 1 summarizes the concept of the adsorptive RLI.

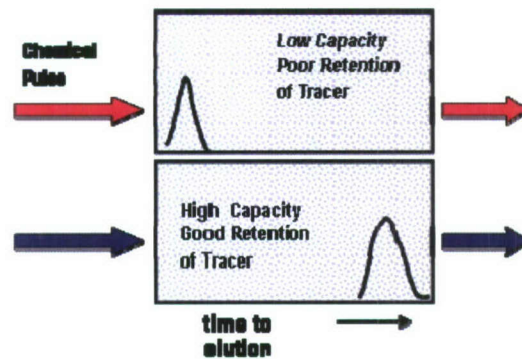


Figure 1. Physical Adsorption Capacity Effect on Retention Time of Chemical Tracer

An ideal tracer chemical for determining the residual adsorptive capacity of a filter has the following properties (Mix and McDonald, 1990 and Smith, 1983):

- Non-destructive and stable (i.e., non-reactive)
- Water soluble
- Volatile; rapid breakthrough for short testing
- Easily detectable
- Easily obtainable
- Relatively non-toxic
- Operable as a leak detector in addition to RLI

The chemical properties of the tracer can be designed to provide information on the integrity of the filter such as the adsorptive or chemical (sorpative) activity of the adsorbent. The selected chemical tracer can provide information on the adsorption capacity of the filter by monitoring the retentivity of the adsorbent for the tracer chemical as illustrated in Figure 1. The retentivity characteristics of the pulse, which include height, width, and retention time, can be correlated to agent or simulant breakthrough curves, which in turn are correlated to an effective remaining residual life capacity of a filter for those agents. Figure 2 illustrates the concept.

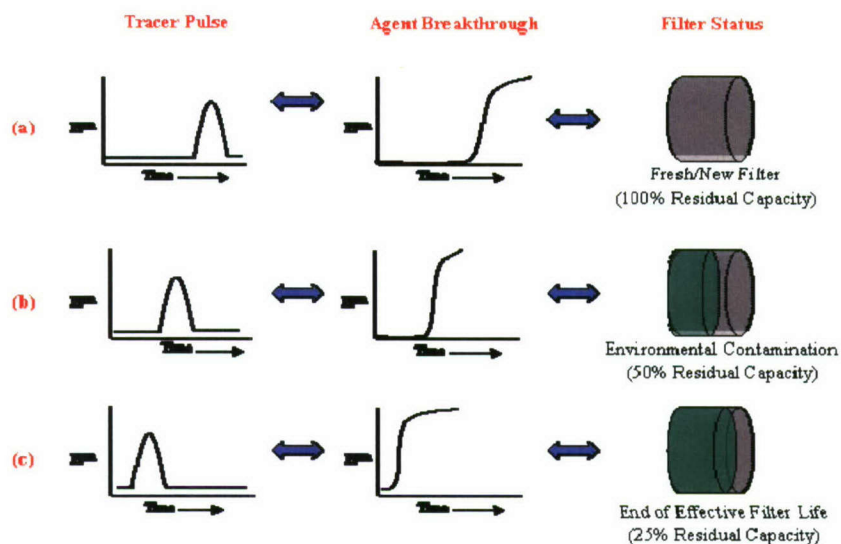


Figure 2. Tracer Pulse and Agent Breakthrough of Contaminated Filters

In addition to environmental contaminants, adsorbed moisture has a large impact on retentivity characteristics of pulsed vapors. As such, tracer compounds must have high solubilities in water. Otherwise, immediate breakthrough will result, and pulses with different contaminations will be indistinguishable from one another.

2.4 Historical Attempts at RLI Development

The pulsed chemical vapor RLI is not a new concept, and several attempts have been made at finding a solution for a non-destructive method of determining residual life of filters. Previous and current methods exist using pulses of freons, alcohols, halogens, and other chemicals to filters. Hammarstrom et al. (1983) reports several other efforts attempting to develop a vapor pulse RLI, including carbon tetrachloride by Turk et al., carbon dioxide by Stamulus and Hammarstrom; low boiling hydrocarbons (e.g., methane, ethane, and propane) by Stamperius and van der Klooster, Hammarstrom et al., Kladnig and Weiss (1978), and freons by Wheat and Hyde, 1972. All of these approaches met with some success; however, each could only be used at low RHs, typically below 30 – 50%. Due to the low solubility of the tracers in water coupled with the relatively high volatility of each tracer, the chemicals were not capable of displacing water; therefore, immediate breakthrough occurred. As such, contaminated and uncontaminated carbon could not be distinguished at high RH conditions.

Karwacki and Morrison (1998) studied pulsing a variety of hydrofluorocarbons (HFCs) and chlorofluorocarbons (CFCs) to carbon beds at a broad range of humidities and met with some success up to a maximum RH of 70%. However, above this humidity, pulses were indistinguishable from leaks; in addition, many chemicals studied were not environmentally friendly.

Mix and McDonald, 1990, reported on the use of methanol as a tracer gas and met with some success at a broad range of RH conditions. Unlike the aforementioned tracer chemicals, the solubility of methanol is such that immediate breakthrough will not occur under

high RH conditions. However, Mix and McDonald were unable to accurately predict residual life based on methanol pulses. Mix also reported on the feasibility of using ammonia as a tracer gas due to its high volatility and water solubility (Mix and McDonald, 1990).

2.5 Tracer Development Approach

Recently, Hunter Applied Research Center (Edgewood, MD) developed a FIT apparatus that addresses the issue of mechanical integrity. This apparatus uses a pulse of acetone to determine if a mechanical leak or other possible flaws are present in a new or in-service filter. Acetone was chosen as a candidate tracer chemical for additional reasons as well; first, acetone is relatively nontoxic and easy to work with. In addition, because of its solubility, acetone is not only retained at high relative humidities, unlike previous FIT tracer chemicals, but the breakthrough and pulse waves vary, depending on the amount of moisture present. It is thought that this may help to determine the amount of water versus the amount of organic loaded on ASZM-T from a fielded system.

Testing was conducted on beds of ASZM-TEDA according to the following methodology:

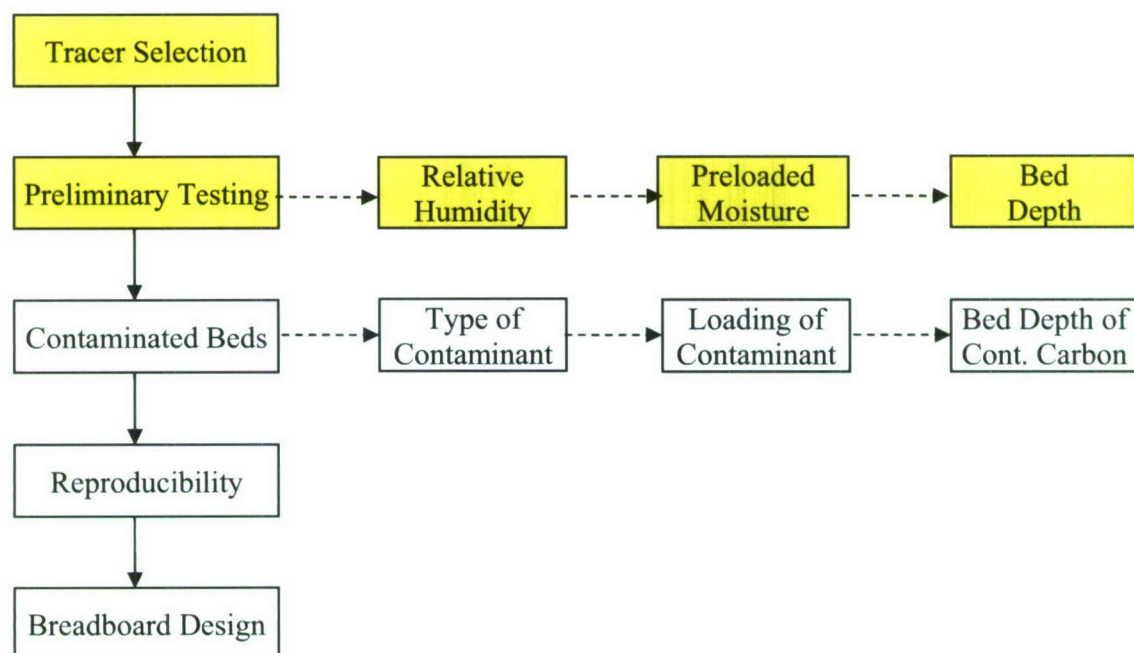


Figure 3. Methodology for Proof-of-Concept Tracer Testing

In an attempt to integrate the Hunter FIT to an RLI, acetone was chosen as a tracer, and pulse testing was subsequently conducted on beds of ASZM-TEDA. This report summarizes preliminary testing, which was conducted at a variety of RH conditions to ensure that the tracer was capable of functioning at the extreme conditions. Various bed depths and challenge concentrations were also examined to determine optimal conditions for trace pulsing.

3. EXPERIMENTAL PROCEDURES

Breakthrough and pulse testing were conducted on a push-pull-vented test apparatus at a temperature of approximately 25 °C. Test tubes were packed using a snowstorm method through a 30-in. drop tube. Carbon samples were humidified in-situ overnight at the specified RH, which was delivered using either a Miller-Nelson control unit or a temperature-controlled saturator cell and measured by an Edgetech dew point hygrometer. The primary conditions for tube testing represented the 100 cfm M48A1 filter, which has a bed depth of approximately 5.2 cm and a log-mean airflow velocity of approximately 22.9 cm/s. These conditions are at a residence time that can also be used to approximate the behavior of the 200-cfm M98 filter.

Acetone was chosen as a candidate tracer because it meets most of the above criteria. Table 1 summarizes the pertinent properties of acetone (NIOSH, 2005), and Table 2 summarizes the pulse test conditions for acetone.

Table 1. Tracer Properties

Property	Values for Acetone
CAS	67-64-1
Molecular Weight	58.1 g/mol
Boiling Point	56.1 °C
Vapor Pressure @ 20 °C	180 mmHg
Water Solubility @ 20 °C	Miscible
8-hr TWA	1000 ppm
IDLH	2500 ppm

Table 2. Pulse Test Conditions

Parameter	Values for Acetone
Challenge Concentration	2,000 mg/m ³
Pulse Duration	5 min
Total Ct	10,000 mg-min/m ³
Airflow Velocity	25.4 cm/s
Bed Depth	5.7 cm
RH	15 - 80%
Pre-Equilibration	Varies
Temperature	25 °C

For acetone feed delivery, a saturator cell, shown in Figure 4, was filled halfway and placed in a temperature-controlled water bath at a set point below room temperature. A stream of nitrogen was passed through the cell, picking up acetone in the vapor phase. This stream was subsequently mixed with a diluent air stream at a rate needed to achieve the desired challenge concentration, which was delivered to the bed of ASZM-TEDA in a square-wave

manner. The acetone feed and effluent concentrations were measured semi-continuously with a Hewlett Packard 5890 Series II GC equipped with a flame ionization detector (FID).

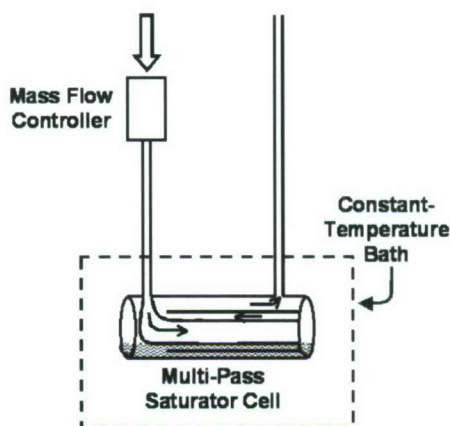


Figure 4. Saturator Cell Used for Feed Delivery

4. RESULTS AND DISCUSSION

4.1 Preliminary Testing

Packed beds of ASZM-T were challenged by flowing a nitrogen stream across a saturator cell half-full of acetone and mixing with a diluent air stream. The feed and effluent concentrations were monitored using a Hewlett Packard 5890 GC equipped with a FID. Preliminary acetone testing was conducted at a challenge concentration of 595 mg/m^3 and included breakthrough and pulse tests. Breakthrough testing was conducted due to a lack of acetone breakthrough data existing on ASZM-TEDA, and the exact breakthrough behavior was unknown. Subsequent acetone pulse testing of carbon beds contaminated with heavy organics was conducted at a challenge concentration of $2,000 \text{ mg/m}^3$ for 5 min.

The first testing conducted consisted of acetone breakthrough testing of beds simulating the M48A1. Testing was conducted at room temperature on 5.2-cm beds of ASZM-T at an airflow velocity of 22.9 cm/s at various RH conditions. Some beds were packed with fresh ASZM-T and tested immediately. Others were pre-equilibrated overnight to quantify the affect of pre-adsorbed moisture on acetone breakthrough. Beds that were not pre-equilibrated are used "as-received" (AR). Those that were equilibrated are denoted with the %RH at which they were equilibrated. For example, 50/80 refers to a test on ASZM-T pre-equilibrated at 50% and conducted at 80% RH. Figure 5 summarizes this testing.

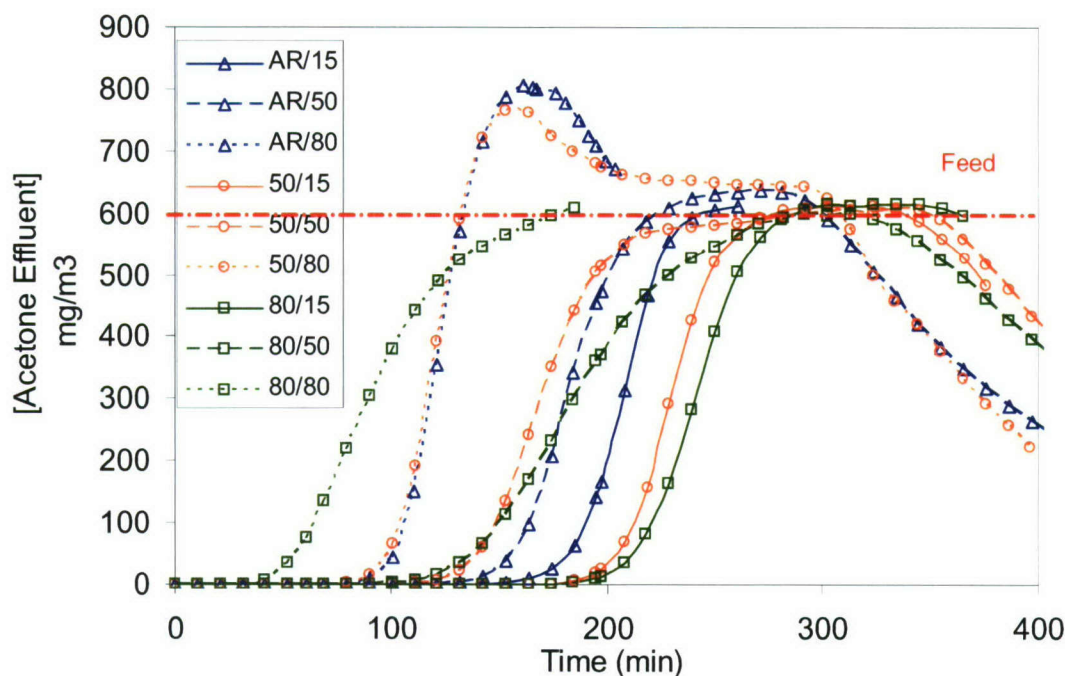


Figure 5. Acetone Breakthrough of ASZM-T at Various RH Conditions.
 $C_0 = 595 \text{ mg/m}^3$, $BD = 5.2 \text{ cm}$, $AFV = 22.9 \text{ cm/s}$.

As Figure 5 shows, acetone breakthrough is clearly affected by the test RH as well as the amount of pre-adsorbed moisture. From the breakthrough curves, it is apparent that those samples tested at 15% RH perform the best against acetone; in addition, pre-adsorbed moisture present on those samples actually enhances the acetone performance, possibly due to the solubility of acetone in liquid water present in the ASZM-T pores. However, once the test RH increases, it is evident that the amount of water present on the carbon causes a decrease in filtration performance. This phenomenon likely occurs because the pre-adsorbed moisture has a higher affinity for additional water as opposed to co-adsorbing with acetone or forming a solution. In addition, water has a significantly lower vapor pressure (i.e., higher boiling point) than acetone.

Another interesting observation in acetone breakthrough behavior is illustrated in the data collected from tests run under 80% RH conditions. Two of the breakthrough curves actually extend beyond the feed concentration; in other words, there is a higher concentration of acetone in the effluent than what was in the feed stream. A possible explanation for this is that acetone is attempting to adsorb on ASZM-TEDA, but being pushed down the bed by the humid air stream, effectively being “rolled-up.” So, at full saturation, there is actually more acetone in the effluent stream due to this roll-up. Evidence of this phenomenon is the fact that once the initial roll-up has occurred, the effluent concentration begins to approach the feed concentration.

A final observation is the difference in the shapes of the breakthrough waves of those tests run under AR conditions compared to those equilibrated at 80% RH. In general, the acetone wavefront under as-received conditions is steeper than the wavefront from equilibrated samples of ASZM-TEDA, indicating a larger mass transfer zone in the humidified samples. This is indicative of internal mass transfer resistances likely stemming from pore diffusion resistance due to the presence of excessive adsorbed moisture.

4.2 Life-Thickness Breakthrough Testing

This preliminary testing revealed that acetone breakthrough was affected by pre-adsorbed moisture as well as test RH, an important factor in determining the residual capacity of a filter. Once this fact was known, the focus shifted to collecting design data for satellite (or sacrificial) filters. A satellite filter will sample the same air as the main filtration system at an equivalent residence time; however, when it comes time to test the residual capacity of the filter, a pulse will be sent through the satellite filter. A more detailed schematic of the satellite filters is described later in the report. The advantages of using satellite filters include some of the following:

- Less chemical tracer is used due to the lower flow rate.
- It is completely non-destructive to the main filter.
- Internal filter components do not have to be either modified or replaced.

Life-thickness breakthrough testing was conducted at a low (AR/15) and high (80/80) RH at a variety of bed depths to establish breakthrough time versus residence time correlations. Figure 6 shows the resulting breakthrough curves at AR/15% RH; whereas, Figure 7 shows acetone breakthrough curves at 80/80% RH.

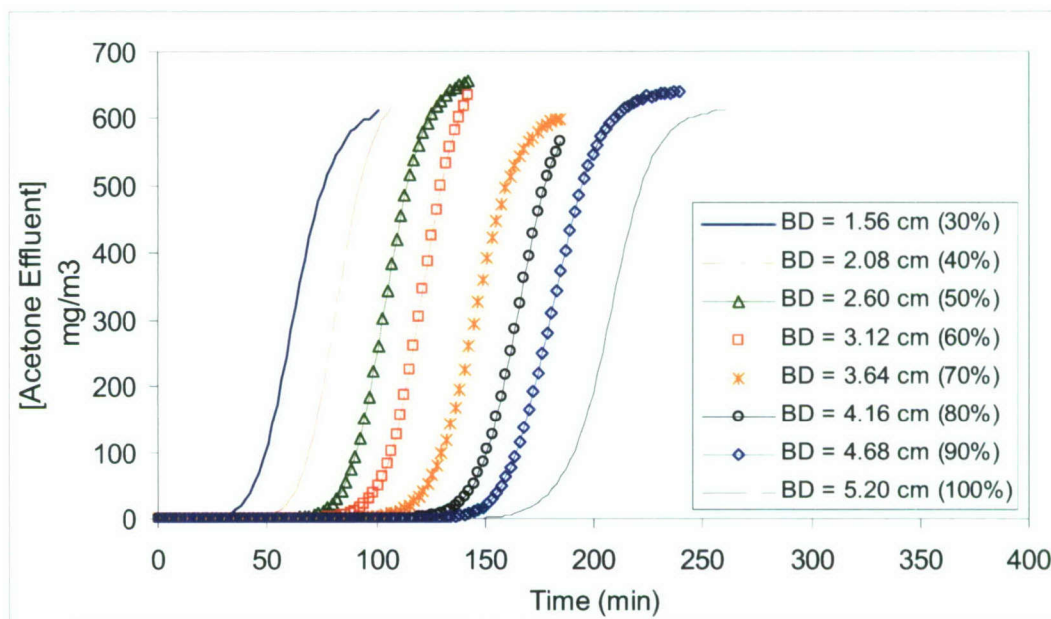


Figure 6. Acetone Breakthrough of ASZM-T at Various Bed Depths.
 $C_0 = 595 \text{ mg/m}^3$, RH = AR/15%, AFV = 22.9 cm/s.

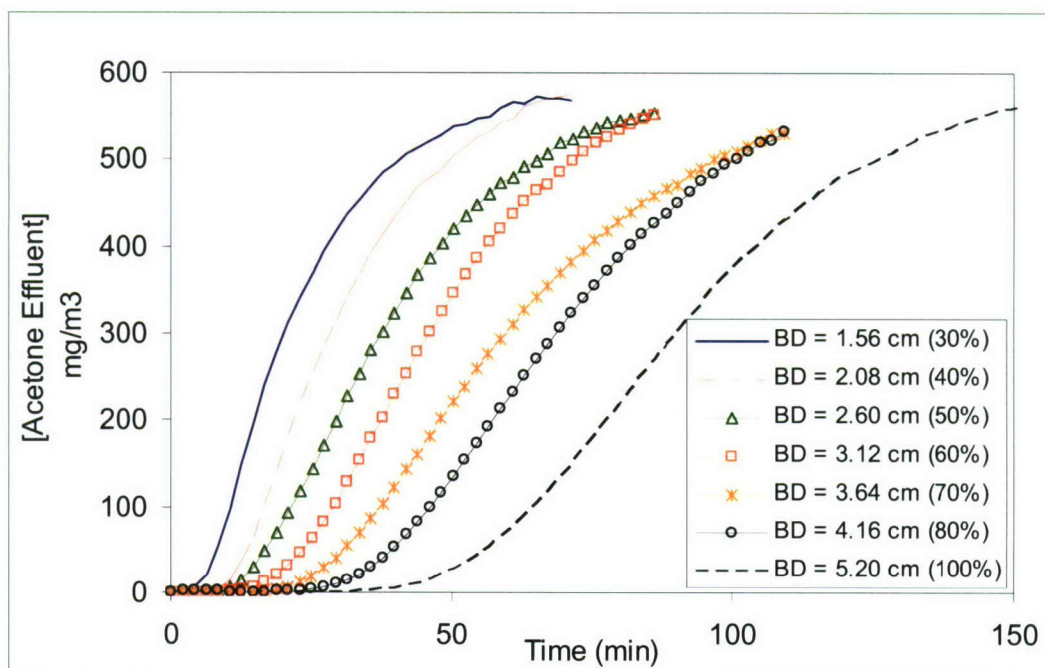


Figure 7. Acetone Breakthrough of ASZM-T at Various Bed Depths.
 $C_0 = 595 \text{ mg/m}^3$, $RH = 80/80\%$, $AFV = 22.9 \text{ cm/s}$.

Results of this testing indicate that it may be possible to use a low residence time (1.56 cm/22.9 cm/s $\sim 0.07 \text{ s}$) for pulse testing. Figure 8 summarizes the correlations developed from the life-thickness testing.

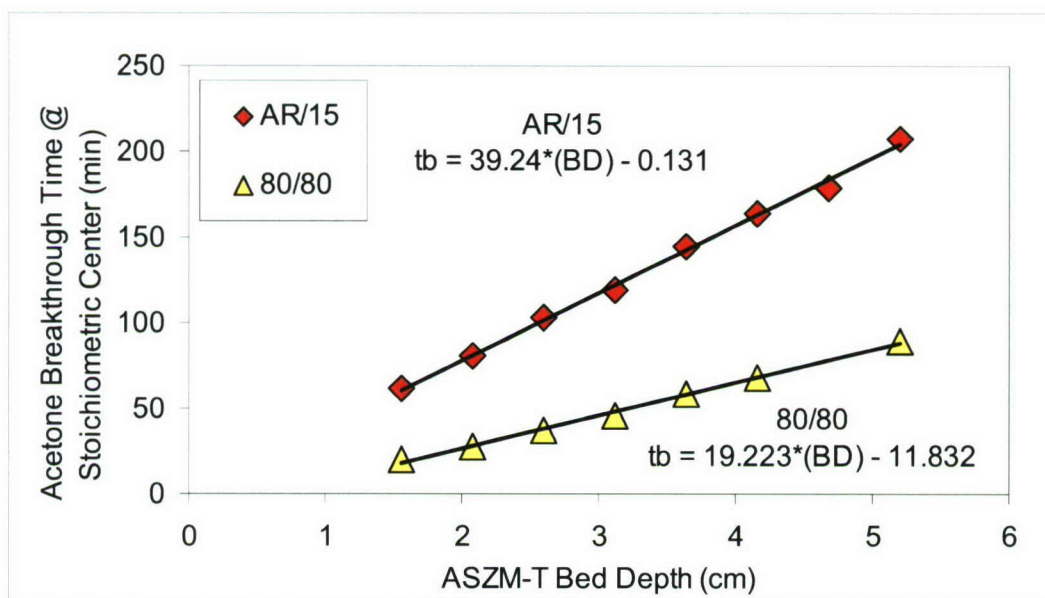


Figure 8. Acetone Breakthrough Time as a Function of ASZM-T Bed Depth.
 $C_0 = 595 \text{ mg/m}^3$, $AFV = 22.9 \text{ cm/s}$.

After completing breakthrough testing at these conditions, testing was conducted by pulsing acetone to the same beds as the ones in Figures 6 and 7. The pulses lasted for 5 min at a challenge concentration of 595 mg/m^3 , equivalent to a dosage (Ct) of approximately $3,000 \text{ mg-min/m}^3$. Figures 9 and 10 illustrate the pulse elution curves through the beds of ASZM-T.

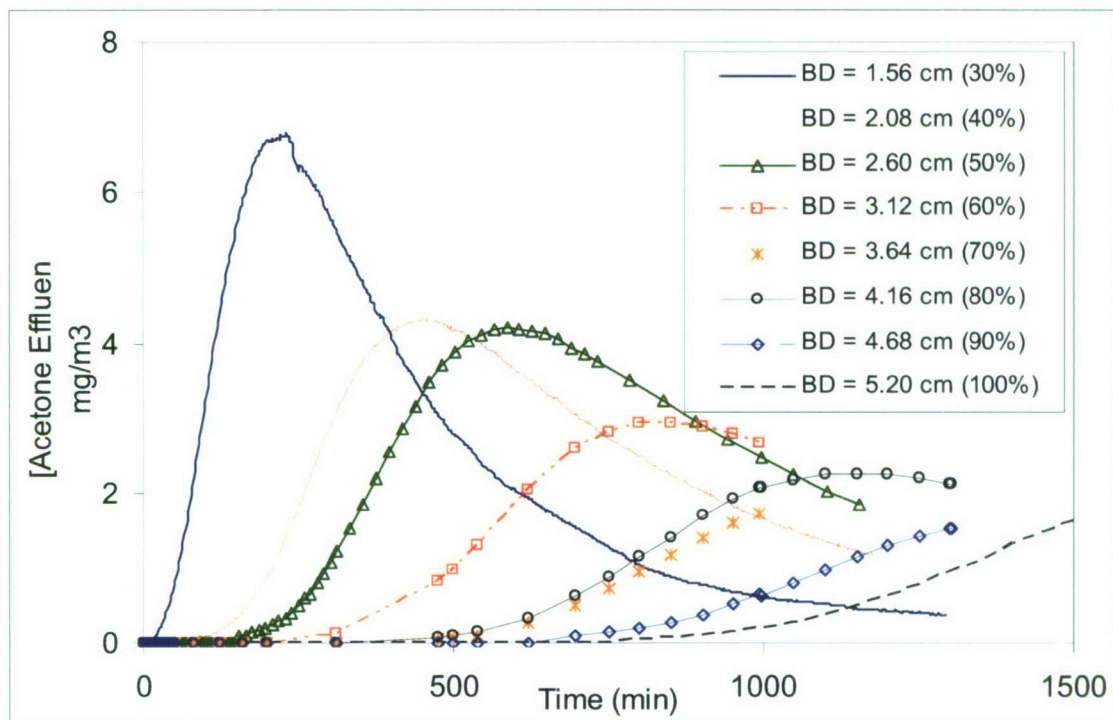


Figure 9. Acetone Pulses to ASZM-T at Various Bed Depths.

$C_0 = 595 \text{ mg/m}^3$ (5 min), RH = AR/15%, AFV = 22.9 cm/s.

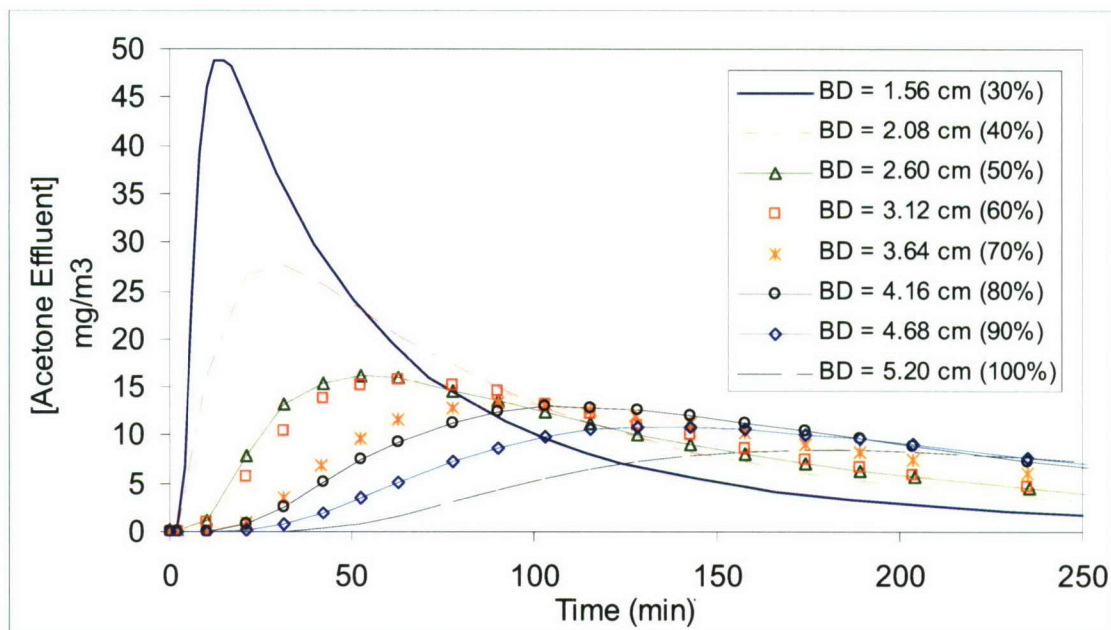


Figure 10. Acetone Pulses to ASZM-T at Various Bed Depths.
 $C_0 = 595 \text{ mg/m}^3$ (5 min), RH = 80/80%, AFV = 22.9 cm/s.

Results from both data sets indicate the possibility of using low residence times to pulse acetone through a satellite filter cartridge. Secondly, the data also show that the same dosage of acetone delivered to a bed of ASZM-T results in different retention (or elution) times and maximum peak heights. For purposes of this report, the retention time is the time it takes the acetone peak to elute from the ASZM-T bed, and the peak height is the maximum concentration that the acetone pulse reaches in the effluent side of the packed bed. Figures 11 and 12 summarize the relationships between acetone peak height, acetone pulse retention time, and ASZM-T bed depth.

Figures 11 and 12 illustrate the quantitative relationships between peak height, peak retention time, and ASZM-T residence time.

4.4 RH and Moisture Content Breakthrough Testing

Based on the life-thickness data, a bed depth was chosen and a more involved study on RH was conducted. Because the elution of the acetone pulse through deep beds takes a very long time, as shown in Figure 10, a bed depth of 2.08 cm was chosen because it provided a reasonable elution time for dry testing, and also held up the acetone pulse at high humidity to differentiate between a leak and residual capacity. Testing was again conducted at room temperature and 22.9 cm/s. Full breakthrough and pulse testing were conducted. Figure 13 shows the breakthrough curves resulting from this testing, and Figure 14 summarizes the data.

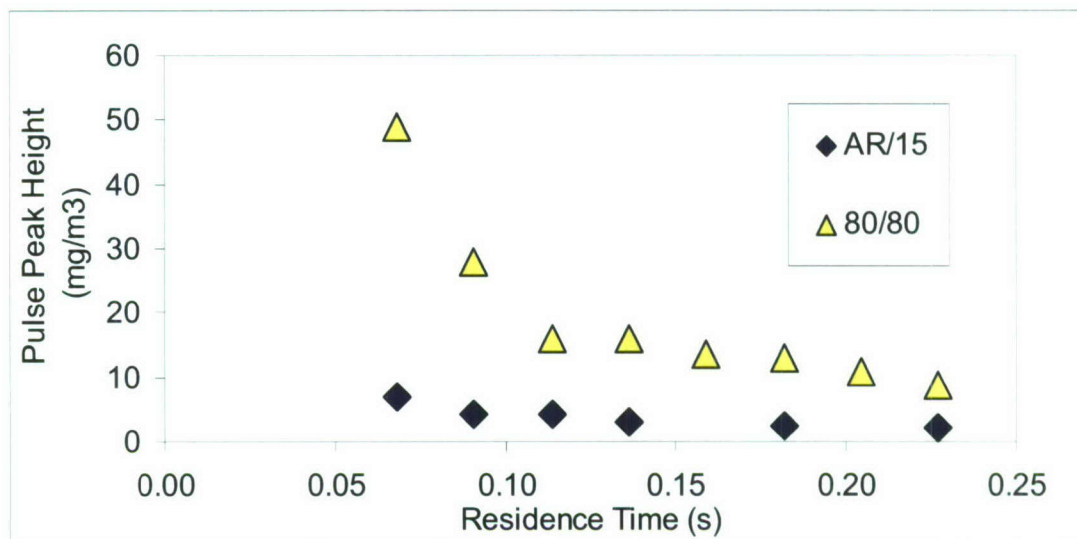


Figure 11. Acetone Effluent Peak Height as a Function of ASZM-T Bed Depth.
 $C_0 = 595 \text{ mg/m}^3$ (5 min), $AFV = 22.9 \text{ cm/s}$.

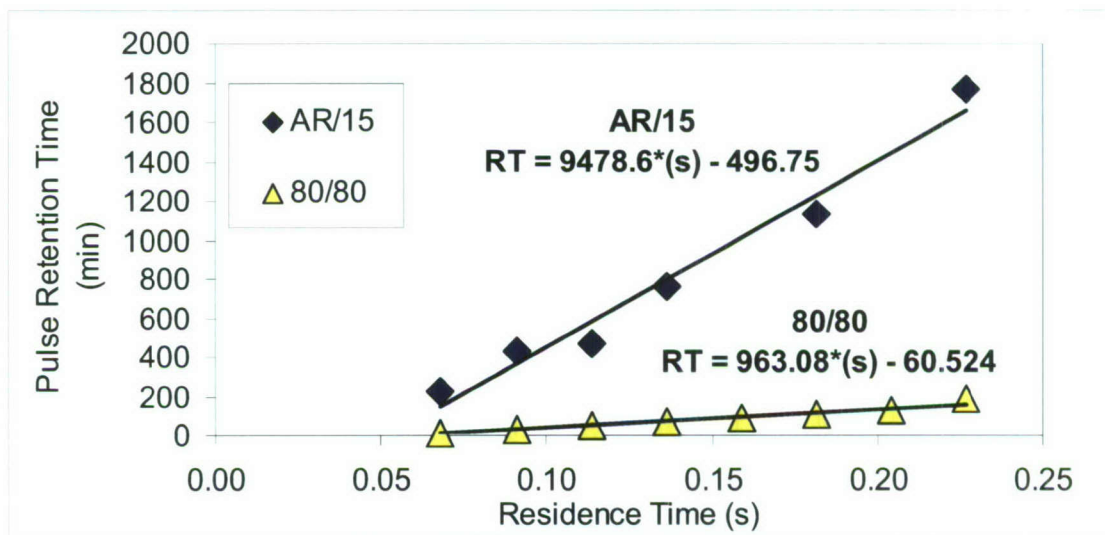


Figure 12. Acetone Pulse Retention Time as a Function of ASZM-T Bed Depth.
 $C_0 = 595 \text{ mg/m}^3$ (5 min), $AFV = 22.9 \text{ cm/s}$.

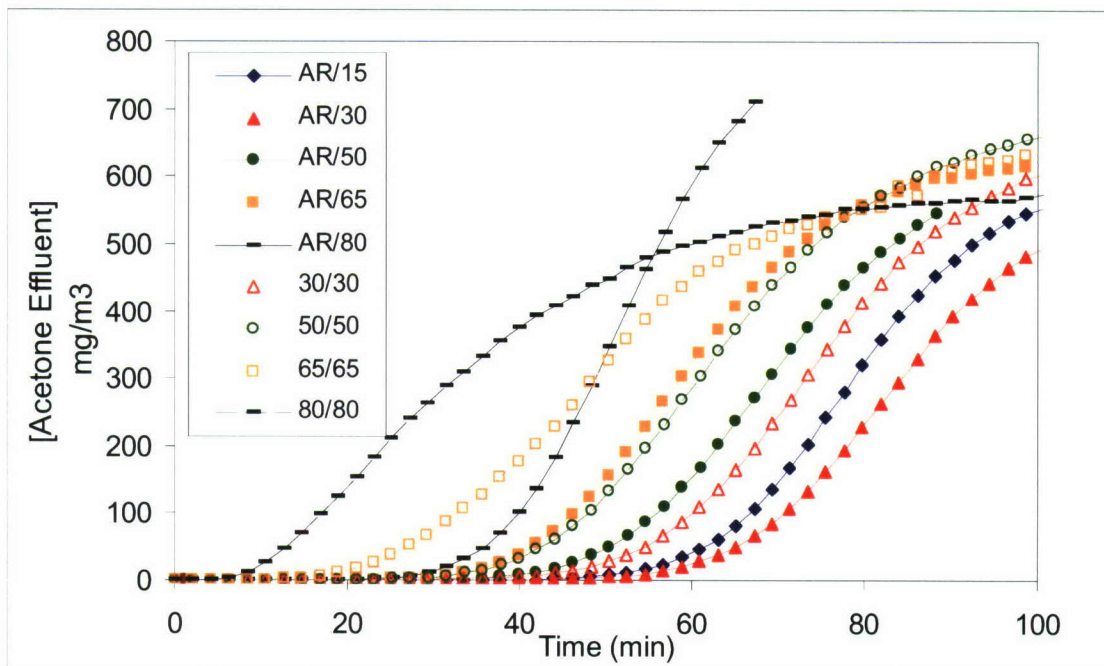


Figure 13. Acetone Breakthrough Time as a Function of RH.
 $C_0 = 595 \text{ mg/m}^3$, $BD = 2.08 \text{ cm}$, $AFV = 22.9 \text{ cm/s}$.

As with the previous testing conducted, as the test humidity increases, the acetone breakthrough time decreases. In addition, as the pre-adsorbed moisture content increases, the breakthrough wave elutes more quickly. Figure 14 summarizes the acetone breakthrough time as a function of RH and moisture content.

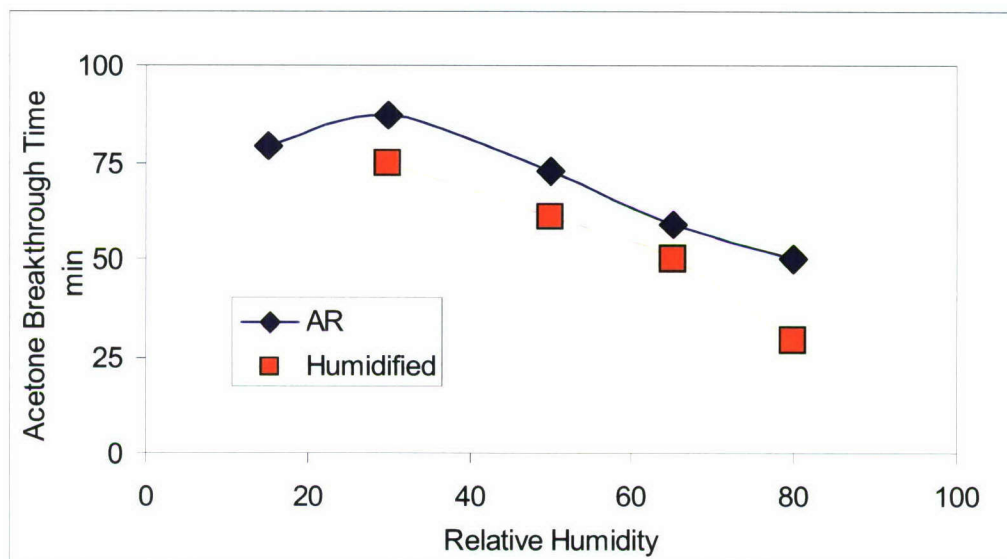


Figure 14. Acetone Breakthrough Time through ASZM-T as a Function of RH.
 $C_0 = 595 \text{ mg/m}^3$, $BD = 2.08 \text{ cm}$, $AFV = 22.9 \text{ cm/s}$.

Figure 14 is a summary of the breakthrough times recorded from the testing conducted at various RH conditions on AR and pre-humidified ASZM-T carbon samples. It appears that an optimum exists between 15 – 30% RH.

4.5 RH and Moisture Content Pulse Testing

Pulse testing was conducted on the same beds and at the same conditions as the breakthrough testing in Figure 13. Figures 15 – 19 summarize the data.

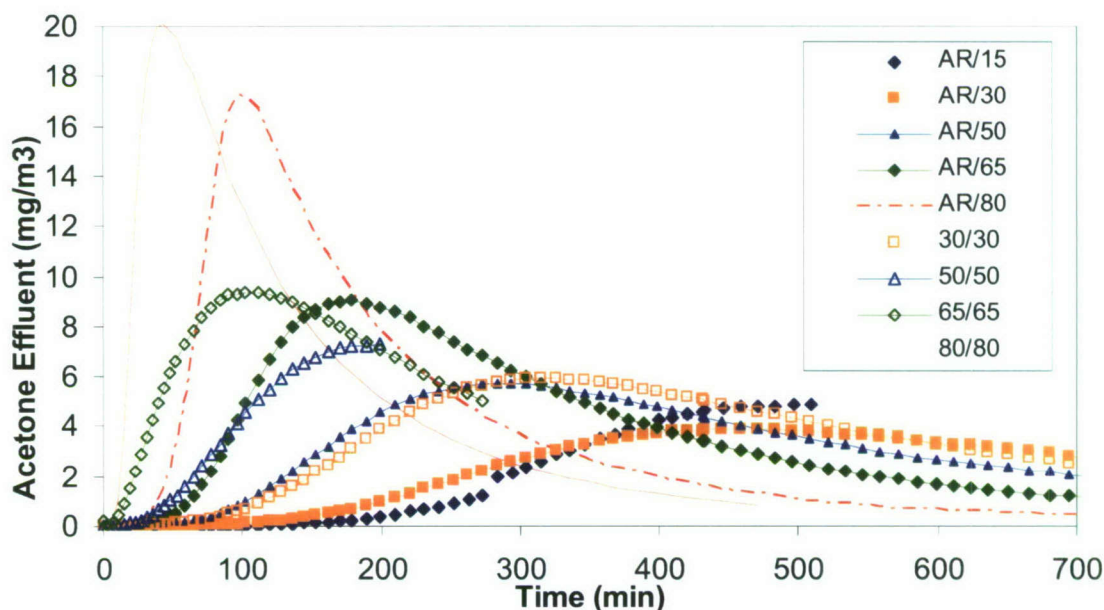


Figure 15. Acetone Pulse Retention as a Function of RH.
 $C_0 = 595 \text{ mg/m}^3$ (5 min), BD = 2.08 cm, AFV = 22.9 cm/s.

Again, as previous testing also showed, pre-adsorbed moisture as well as test humidity affects the shape and elution time of the acetone pulse. A higher RH and pre-adsorbed moisture content leads to shorter elution times as well as higher elution concentrations. In addition to differentiating between RHs, it is desired to differentiate between samples that are preloaded with water and those that are not. Figure 16 illustrates the pulses at 30% RH.

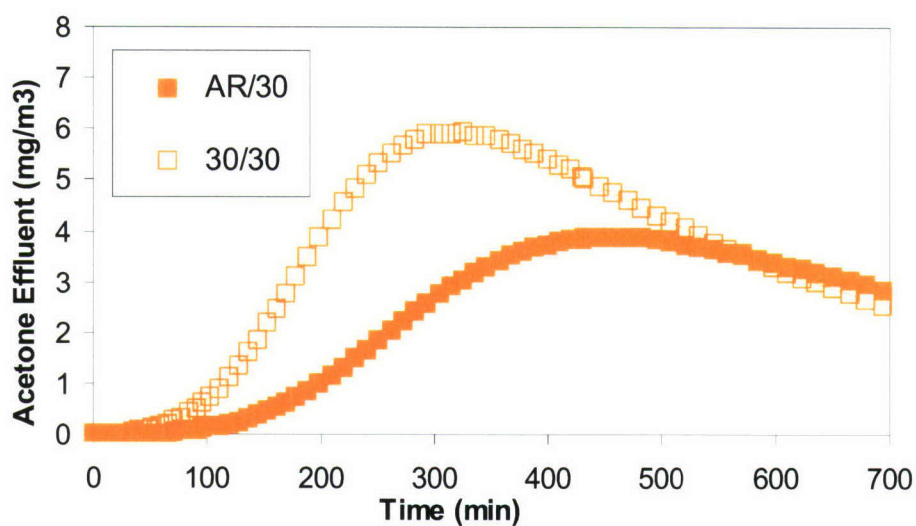


Figure 16. Acetone Pulse Retention as a Function of 30% RH.
 $C_0 = 595 \text{ mg/m}^3$ (5 min), BD = 2.08 cm, AFV = 22.9 cm/s.

The pulses at 30% RH show that samples with pre-adsorbed moisture elute faster and have a higher elution concentration. Over a long period of time (~1 day), all of the acetone will elute off of the filter and is therefore not detrimental to filter performance.

Pulses at 50% RH show similar trends to those at 30% RH, as shown in Figure 17.

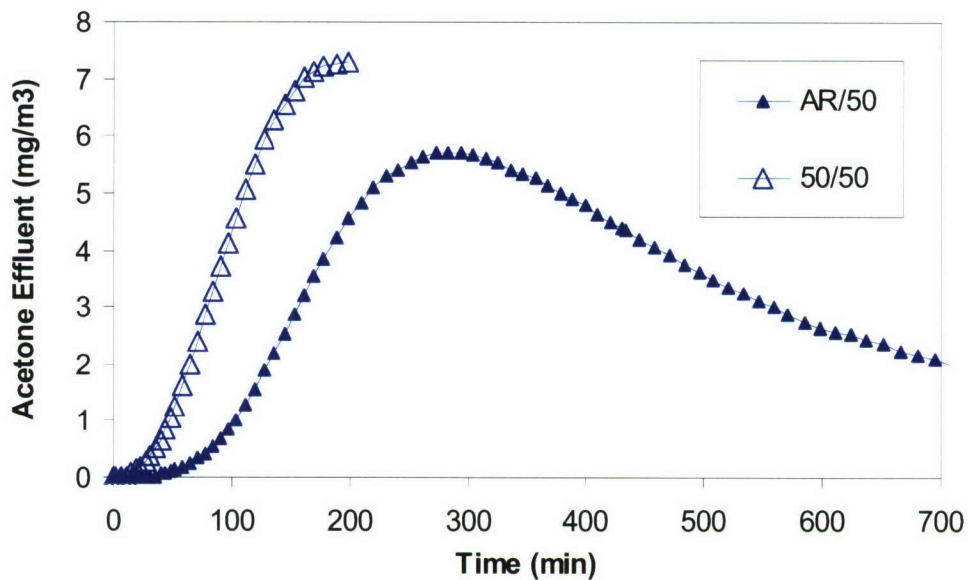


Figure 17. Acetone Pulse Retention as a Function of 50% RH.
 $C_0 = 595 \text{ mg/m}^3$ (5 min), BD = 2.08 cm, AFV = 22.9 cm/s.

Again, data show that samples with pre-adsorbed moisture will elute quicker and at a higher concentration than AR samples. Also, note that these samples elute faster than those at 30% RH as well as at a higher concentration.

Pulses at 65% RH were conducted and again show similar results and trends as those measured at previous RH and moisture loadings. Figure 18 illustrates the acetone pulse curves through ASZM-T.

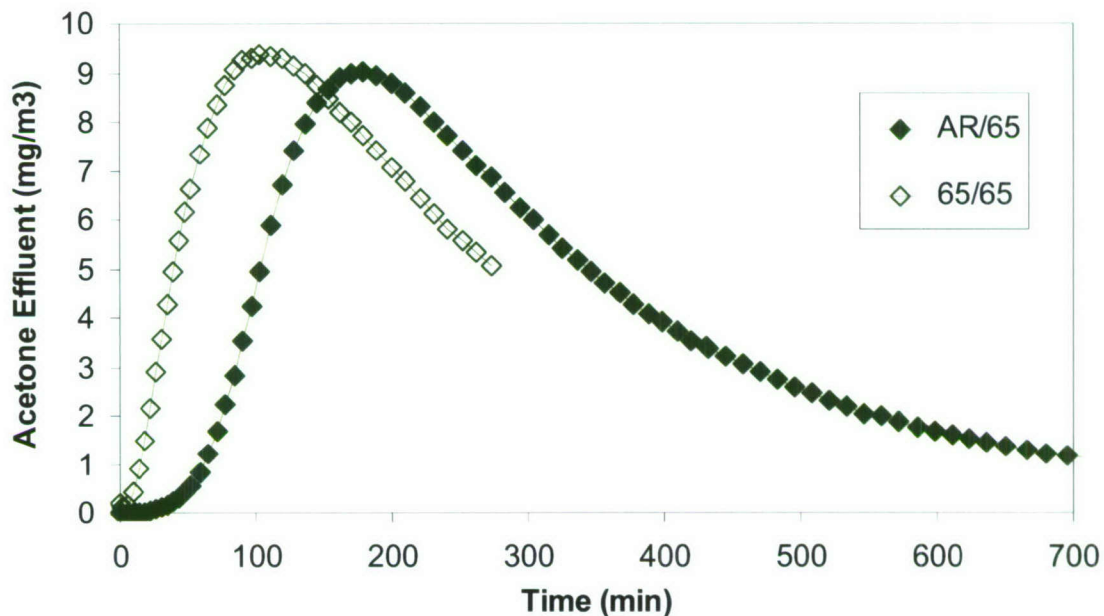


Figure 18. Acetone Pulse Retention as a Function of 65% RH.
 $C_0 = 595 \text{ mg/m}^3$ (5 min), BD = 2.08 cm, AFV = 22.9 cm/s.

As with the previous samples, pre-adsorbed samples elute quicker and at a higher concentration. Both samples at 65% RH also elute quicker and at a higher concentration than those at 30 or 50% RH.

As shown in Figure 19, the final samples were run at 80% RH..

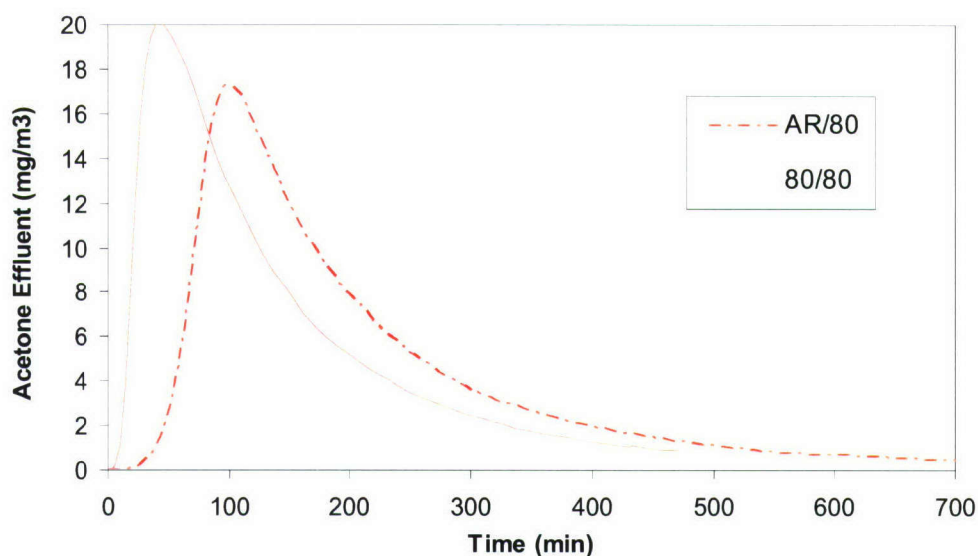


Figure 19. Acetone Pulse Retention as a Function of 80% RH.
 $C_0 = 595 \text{ mg/m}^3$ (5 min), BD = 2.08 cm, AFV = 22.9 cm/s.

As with all previous samples tested, the pre-adsorbed sample elutes first and at a higher concentration. Each sample tested resulted in a unique pulse elution wave at a specific elution time and at a specific elution concentration. It is the objective of this effort to create correlations describing the relationships between these parameters, and to use these relationships to estimate the residual life capacity of an in-service filter.

Figures 20 and 21 summarize the data collected from the pulses at various RH conditions.

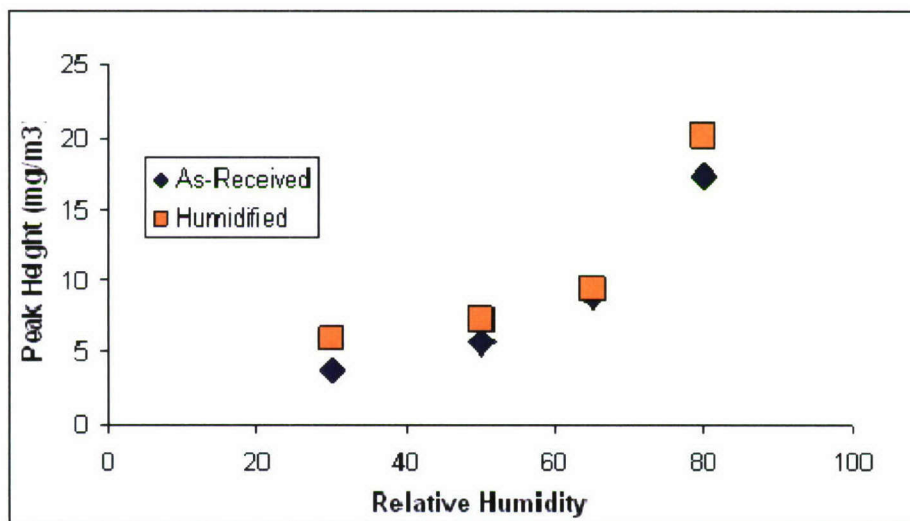


Figure 20. Elution Peak Height of Acetone Pulses as a Function of RH.
 $C_0 = 595 \text{ mg/m}^3$ (5 min), BD = 2.08 cm, AFV = 22.9 cm/s.

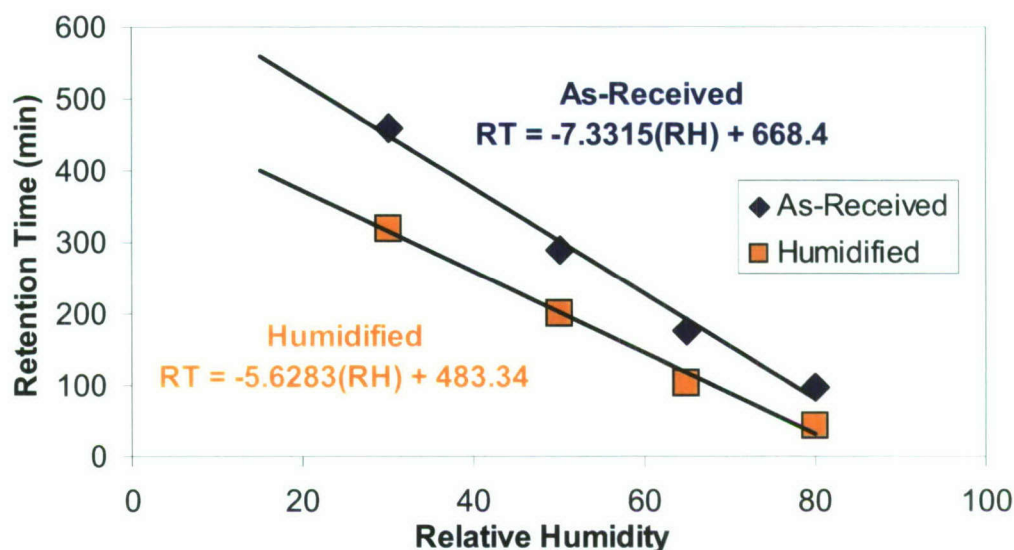


Figure 21. Acetone Pulse Retention Time as a Function of RH.
 $C_0 = 595 \text{ mg/m}^3$ (5 min), BD = 2.08 cm, AFV = 22.9 cm/s.

Figure 20 shows that above 65% RH, much more acetone is eluted. This finding is consistent with the water isotherm, which shows that moisture content on ASZM-T increases dramatically above approximately 65% RH. It is likely that more acetone elutes more quickly at a higher RH because of its lower boiling point relative to water.

The results in Figure 21 demonstrate that a linear relationship exists between the elution time of the acetone pulse and the RH and moisture content of the sample over the RH range studied, which makes establishing correlations an easier problem.

5. CONCLUSIONS

Previous attempts to develop a vapor pulse method for determining the residual physical adsorption capacity of NBC filters were investigated. The properties a tracer must possess to effectively determine the residual life of an NBC filter were established, and acetone was chosen as a candidate. Breakthrough and pulse testing were conducted on various bed depths of ASZM-TEDA containing a range of moisture loadings at a variety of relative humidity (RH) conditions. The following trends, observations, and conclusions were drawn from the data collected:

- Acetone breakthrough and pulse retention times through ASZM-T are linear over a wide range of residence times; similarly, this would allow easy scaling of a satellite cartridge.
- All of the acetone loaded on ASZM-T desorbs over time.

- Increasing the test RH decreases the breakthrough time and pulse elution time of acetone through ASZM-T.
- In general, increasing the pre-adsorbed moisture content on ASZM-T typically results in a decrease in either breakthrough or pulse elution time and increases the peak height of the acetone pulse.
- The acetone pulse retention time through ASZM-T is linear with RH.
- The acetone elution peak height is non-linear with RH and seems to be dependent on the water isotherm.
- Acetone may be a good candidate tracer because different test RH conditions and preloaded moisture contents are distinguishable based on the retentivity of the pulse.
- Acetone may be a poor candidate tracer due to the time it takes for a pulse to elute through the bed.

Based on the conclusions, a higher concentration of acetone will be pulsed to beds contaminated with heavy organics. Testing will be conducted at a variety of RH conditions to determine if acetone pulse behavior is capable of distinguishing between different contaminants, different contamination levels and moisture contents.

LITERATURE CITED

Favas, G. *End of Service Life Indicator (ESLI) for Respirator Cartridges. Part I. Literature Review*; DSTO-TN-0657; Defence Science and Technology Organisation, Fishermans Bend, Victoria, Australia, 2005.

Gardner, P. et al. *Development and Design of a Colorimetric End-of-Service-Life Indicator*; ECBC-CR-065; U.S. Army Edgewood Chemical Biological Center: Aberdeen Proving Ground, MD, 2003; UNCLASSIFIED Report (AD-B298 053).

Mix, T.W.; McDonald, T.C. *A Vapor Challenge Method of Measuring the Residual Life of Gas Filters*; CRDEC-CR-086; U.S. Army Chemical Research, Development and Engineering Center: Aberdeen Proving Ground, MD, 1990; UNCLASSIFIED Report (AD-277 711).

Smith, M.E. Master of Philosophy Thesis, Chemical Defence Establishment, Porton Down, Salisbury, Wilts, 1983.

Hammarstrom, J. et al. *Residual Life Method for Determining Gas Protection of ASC Whetlerite Carbon Beds*; ARCSL-CR-83046; U.S. Army Chemical Systems Laboratory: Aberdeen Proving Ground, MD, 1983; UNCLASSIFIED Report (AD-B079 938).

Turk, A.; Makr, H.; Mehlmann, S. Tracer Gas Nondestructive Testing of Activated Carbon Cells. *Mat. Res. Stand.* **1969**, 9, 24.

Stamperius, P.C.; Van der Klooster, H.W. Leaks in Charcoal Beds and the Water Content of Charcoal Determined Using the Ethan Pulse Test Technique. *Chem. Lab. TNO, Nat. Def. Res. Org., Rijswijk, Netherlands*, **1973**, 9.

Stamulis, A.; Thompson, J.K.; Bogardus, H.F. Carbon Dioxide Pulse Technique for Determining the Condition of Adsorbent Charcoal Beds. *J. Air Poll. Assoc.* **1971**, 21, 709.

Kladnig, W.F.; Weiss, A.H. *Residual Protective Life of Carbon Beds*; Quarterly Progress Report No. 9; Worcester Polytechnic Institute: Worcester, MA, June 1978 – August 1978; UNCLASSIFIED Report.

Wheat, J.A.; Hyde, J.C. *Estimation of the Residual Adsorption Capacity of Charcoal Filters*; Defence Research Establishment Ottawa Report No. 663; National Defence Headquarters, Ottawa, Ontario, Canada, 1972; UNCLASSIFIED Report.

Karwacki, C.J.; Morrison, R.W. Adsorptive Retention of Volatile Vapors for Nondestructive Filter Leak Testing. *Ind. Eng. Chem. Res.* **1998**, 37, 3470-3480.

NIOSH Pocket Guide to Chemical Hazards, <http://www.cdc.gov/niosh/npg>. NIOSH Publication No. 2005-151; Washington, DC, 2005.